

## AMENDED SPECIFICATION

Reprinted as amended in accordance with the Decision of the Assistant-Comptroller, acting for the Comptroller-General, dated the ninth day of May, 1947, under Section 21 of the Patents and Designs Acts, 1907 to 1946.

## PATENT SPECIFICATION

563,481



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## PROVISIONAL SPECIFICATION

### Improved Process for the Alcoholysis of Oils

We, VICTOR WOLF LIMITED, a British Company, of Victoria Works, Croft Street, Clayton, Manchester, 11, and RICHARD ROWE, a British Subject, of the Company's address, do hereby declare the nature of this invention to be as follows:—

This invention relates to the alcoholysis of vegetable and animal oils such as castor oil, fish and whale oils, linseed oil and cocoanut oil, (hereinafter compendiously referred to as oils) and is concerned with such alcoholysis when carried out in the presence of an acid, e.g. hydrochloric or sulphuric.

The alcoholysis of castor oil by hydrochloric acid has so far been carried out in such a way that a mixture of oil and alcohol with hydrochloric acid was heated under reflux for several hours, that is to say from 4 to 5 hours, the quantity of alcohol always exceeding that of the oil. For example, in the alcoholysis of castor oil by methyl alcohol, the quantity of alcohol used was at least 130 parts to 100 parts castor oil, that is 12.5 times the amount of alcohol required by theory.

By research and experiment we have found that if the amount of alcohol used is less than five times the amount required by theory, and preferably between about 1.5 and 3.5 times the quantity required by theory, (that is for example 25 parts of methyl alcohol to 100 parts of castor oil), then not only is there the economy resulting from the use of the smaller quantity of alcohol but the main part of the glycerine may separate in a lower layer, especially if methyl or ethyl alcohol be used. Elevated tem-

peratures (by which we do not refer to the warming requisite to make a fat like cocoanut oil fluid) have moreover been found to be unnecessary, even with such relatively small proportions of alcohol. The lower temperatures obviate the danger of formation of chlorhydrin and allow of the simplification of the apparatus used. In addition, there is an advantage over the alkaline alcoholysis process in that oils may be used as starting material that contain free fatty acids, that is without previous refining.

Our invention comprises the use in the acid alcoholysis of oil of a quantity of alcohol less than five times the amount required by theory, and preferably between 1.5 to 3.5 times the quantity of alcohol theoretically required.

Our invention further comprises the use of normal temperatures in the alcoholysis.

The amount of acid used in the process is preferably between 1 and 3% of the mixture, but if the process is carried out at normal temperatures, the amount of acid used need not be so limited.

In carrying out the process, after the conclusion of the alcoholysis, the lower layer (if any) containing glycerine together with alcohol is removed and after neutralization and distillation of the alcohol, it is worked up in the usual way to isolate the glycerine. The mixture of the esters is repeatedly washed with water to remove the entire or the remaining glycerine and alcohol. The wash water may be acidified or made alkaline as required.

If a special value is attached to great purity or isolation of particular fractions

[Price 2/-]

of esters, as may be determined by the intended use of the esters, they may be distilled *in vacuo*.

#### EXAMPLES.

- 5 12 parts hydrochloric gas are introduced into a mixture of 500 parts Castor Oil (A.V.6) and 125 parts methanol. Soon turbidity is observed, and after a few, say 3 to 4 hours, a glycerine layer has settled down. It is removed and the oily layer washed several times with little water which takes off the remaining glycerine, methanol and acid. When heated, the water content of the oily layer settles down and can be drawn off.

The oil is then distilled under a vacuum of 3 mm. The distillate consists of the methyl-esters of the Castor Oil fatty acids; an eventual residue may be used for the next alcoholysis. The glycerine layer, by itself or together with the wash-water, after separation from floating oil is neutralised with a solution of alkali-hydroxide and the methanol distilled off. Eventually the glycerine is recovered from the salt solution by concentration and distillation.

Dated this 7th day of January, 1943.

MARKS & CLERK.

### COMPLETE SPECIFICATION

#### Improved Process for the Alcoholysis of Oils

We, VICTOR WOLF LIMITED, a British Company, of Victoria Works, Croft Street, Clayton, Manchester, 11, and RICHARD ROWE, a British Subject, of the Company's address, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the alcoholysis of vegetable and animal glyceride oils such as castor oil, fish and whale oils, linseed oil and cocoanut oil, (hereinafter compendiously referred to as natural oils or oils) and is concerned with such alcoholysis when carried out in the presence of hydrochloric acid.

The alcoholysis of castor oil by hydrochloric acid has so far been carried out in such a way that a mixture of oil and alcohol with hydrochloric acid was refluxed under anhydrous conditions for several hours, that is to say from 4 to 5 hours. The quantity of alcohol always exceeded that of the oil which means in the case of alcohols having less than 6 carbon atoms, an enormous excess over the amount needed by theory. For example, in the alcoholysis of castor oil by methyl alcohol, the quantity of alcohol used was at least 130 parts to 100 parts castor oil, that is 12.5 times the amount of alcohol required by theory.

By research and experiment we have found that the process of alcoholysis of oils in the presence of hydrochloric acid can be carried out at room temperatures which avoids all the complications and difficulties connected with the use of hot or boiling hydrochloric acid, including the limited range of suitable materials for the apparatus and the danger of formation of chlorohydrin.

We have further found that the process can be carried out even at room temperatures with amounts of alcohol much less than has heretofore been considered necessary, and as low as between 1.5 to 3.5 times the quantity required by theory (that is, as an example, 25 parts of methyl alcohol to 100 parts of castor oil). This results in a very considerable economy in the process. In addition, especially when methyl or ethyl alcohol is used, the main part of the glycerine separates in a lower layer. In certain alkaline alcoholysis processes, this separation of glycerine can be achieved, but in such processes oils containing free fatty acids cannot be used as in our process.

We have further found that the process can be carried out most advantageously if alcohols are used having less than 6 carbon atoms and the best results will be achieved if the original oil, or at least the desired alkyl esters of the fatty acids, are soluble in the alcohol used in the alcoholysis at the reaction temperature.

Our invention consists in a process for the alcoholysis of natural oils as hereinbefore defined, comprising treating the oil with an alcohol having less than six carbon atoms in the presence of hydrochloric acid at room temperatures apart from any warming that may be necessary to make the oil fluid. The process is carried out preferably under anhydrous conditions.

Our invention further comprises the use in the acid alcoholysis of oil at room temperatures (apart from any warming that may be necessary to make the oil fluid) of a quantity of alcohol less than five times the amount required by theory, and preferably between 1.5 to 3.5

times the quantity of alcohol theoretically required.

The amount of hydrochloric acid used in the process is preferably between 1 and 3% of the mixture. The gaseous acid is introduced preferably into the alcohol or into the mixture of the oil with the alcohol.

When the alcoholysis is complete or substantially complete, the reaction product can be worked up in the following manner:—the lower layer (if any) containing glycerine together with excess alcohol is removed and after neutralisation and distillation of the alcohol, it is worked up in the usual way to isolate the glycerine. The mixture of the esters is repeatedly washed with water (made alkaline if desired) to remove the remaining glycerine and alcohol. If special value is attached to great purity or isolation of particular fraction of esters, as may be determined by the intended use of the esters, they may be distilled *in vacuo*.

#### EXAMPLES.

1. 12 parts hydrochloric gas are introduced into a mixture of 500 parts Castor Oil (A.V.6) and 125 parts methanol (2.5 equivalents). Soon turbidity is observed, and after a few, say 3 to 4 hours, a glycerine layer has settled down. It is removed and the oily layer washed several times with little water which takes off the remaining glycerine, methanol and acid. When heated, the water content of the oily layer settles down and can be drawn off. The oil is then distilled under a vacuum of 3 mm. The distillate (480 parts boiling up to 225° C.) consists of the methyl-esters of the Castor Oil fatty acids; an eventual residue may be used for the next alcoholysis. The glycerine layer, by itself or together with the washwater, after separation from floating oil is neutralised with a solution of alkali-hydroxide and the methanol distilled off. Eventually the glycerine is recovered from the salt solution by concentration and distillation.

2. 27 parts hydrochloric acid are introduced into 400 parts propyl alcohol (3 equivalents). The solution is stirred with 500 parts Coconut Oil for several hours, whilst the temperature is kept above its melting point. After cooling down, the bottom layer, consisting mainly of glycerine, is removed. The oily layer is washed several times with water, in order to remove the remaining glycerine, the excess of propyl alcohol, and the hydrochloric acid. The oil is

then heated and the water which settles down, drawn off. Finally, the product is distilled in vacuum. The colourless distillate, 480 parts, boiling from 130 to 200° C. at 3 mm. consists of the propyl esters of the coconut fatty acids (S.V. 219). The glycerine layer is worked up according to Example 1.

3. 100 parts Castor Oil are mixed with 60 parts butyl alcohol (2.5 equivalents). 3.2 parts hydrochloric acid are introduced into the mixture. After a few days the glycerine, the surplus of butanol and the hydrochloric acid are removed by repeated stirring with water. The oil after drying, is distilled until a boiling point of 255° C. is reached in a vacuum of 2½ mm. The distillate, which corresponds in weight approximately to the raw material used, is a clear yellowish oil and has a Sap. Value of 160 (Sap. Value of the butyl esters of ricinoleic acid = 158).

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the alcoholysis of natural oils as hereinbefore defined, comprising treating the oil with an alcohol having less than six carbon atoms in the presence of hydrochloric acid at room temperatures apart from any warming that may be necessary to make the oil fluid.

2. A process for the alcoholysis of natural oils as hereinbefore defined, comprising treating the oil at room temperatures (apart from any warming that may be necessary to make the oil fluid) in the presence of hydrochloric acid, with an alcohol having less than six carbon atoms in amount less than five times that required by theory.

3. A process for the alcoholysis of natural oils as hereinbefore defined, comprising treating the oil at room temperature (apart from any warming that may be necessary to make the oil fluid), in the presence of hydrochloric acid, with between 1.5 and 3 equivalents of an alcohol having less than 6 carbon atoms.

4. A process as claimed in claims 1—3, in which the natural oil used is castor oil.

5. Alkyl esters of fatty acids of animal or vegetable oils, whenever produced by the methods hereinbefore described and claimed.

Dated this 5th day of January, 1944.  
MARKS & CLERK.

1. The first step is to identify the problem or question that needs to be answered. This involves understanding the context and the specific requirements of the task.

2. Next, gather relevant information and data. This may involve research, consultation with experts, or collecting data from various sources.

3. Once the information is gathered, analyze it to identify patterns, trends, and potential solutions. This step often involves critical thinking and problem-solving skills.

4. After analysis, develop a plan or strategy to address the problem. This plan should outline the steps to be taken and the resources needed.

5. Implement the plan and monitor progress. This involves putting the plan into action and regularly checking on the results to ensure that the problem is being solved effectively.

6. Finally, evaluate the outcome and make adjustments as needed. This step involves reflecting on the process and the results to determine what worked well and what could be improved for future tasks.

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SECRET

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1. The first step in the process of the investigation is to identify the problem or issue that needs to be addressed. This involves gathering information about the situation and determining the scope of the investigation.